Optimization of the thermal performance of PCM nanocomposites

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Thermal energy storage is among the highly efficient approaches to overcome the energy crisis. Using phase change material (PCM) is one of the most effective techniques in thermal energy storage applications. Several types of PCM with distinct characteristics and different ranges of melting and solidification temperature have found their way in various industries. However, commercialized PCMs generally suffer from low thermal conductivity which limits their application. In this study, the effect of adding different weight percentages of various nanoparticles, such as CuO, TiO_2, Al_2O_3 and graphene to paraffin, as a standard PCM, on the improvement of the thermal properties of PCM was investigated. Thermophysical properties and morphology of the nanocomposites, such as phase change temperature and latent heat of melting were characterized by Differential Scanning Calorimetry (DSC), Scanning Electron Microscope (SEM), and Fourier Transform Infrared Spectroscopy (FT-IR). SEM images display the proper distribution of nanoparticles in phase change material. FTIR results verified the formation of nanocomposites. A comparison between the investigated PCM nanocomposites showed that the nanocomposites containing 2 wt.% TiO_2 with the enthalpy of 179.88 J/g, and 1 wt.% graphene nanocomposite with the enthalpy of 120.38 J/g had the highest and lowest energy storage capacity compared to paraffin, respectively. The results indicated that Nano-enhanced phase change materials (NEPCMs) could be particularly useful in applications in which temperature control is crucial. The new types of nanocomposites used in this study showed remarkable thermal performance, and they are capable of being used in thermal management applications. © 2020 Journal of Energy Management and Technology

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1. INTRODUCTION

Global warming is one of the most crucial challenges in the current century, and controlling the emission of greenhouse gases is also one of the first steps to control global warming [1]. The need to use clean and renewable means of energy, such as thermal energy, is of vital importance to fulfill the increased demand for energy and also to control environmental contamination [2]. Phase change materials (PCMs) are widely used as an effective and economical means of thermal energy storage [3, 4]. Phase change materials are assessed by their capacity for thermal energy storage (TES) and their thermal conductivity. Latent heat and thermal conductivity of PCMs are the limiting factors governing the efficiency of TES systems. However, the thermal conductivity of typical commercially available PCMs is considerably low. Besides, expensive heat transfer systems are frequently employed in TES systems to achieve high rates of heat charge/discharge and to improve the efficiency of the system. Therefore, the enhancement of the heat transfer rate has a crucial role in TES applications. Hence, different materials, such as metal/metal oxide nanoparticles and carbon fibers, can be added to the PCM to improve its heat transfer properties [2–5]. Paraffin is one of the most important types of organic PCMs. Generally, the melting point and heat of fusion of paraffin increase as the carbon chain length increases. Paraffin is used widely in energy storage applications because they are chemically inert and stable at high temperatures. Furthermore, they have a slight volume change during melting, and their vapor pressure is low in the

liquid form. Therefore, systems that use paraffin as the energy storing material are durable, non-corrosive, and inexpensive. Melting and freezing point of linear hydrocarbons with 13 to 18 carbons are in the range of 5.5 to 61.4 °C [6]. Despite some desirable characteristics, such as congruent melting and proper nucleation, paraffin shows some unfavorable properties, such as low thermal conductivity, incompatibility with plastic materials, and partial flammability [7]. Saeid Al-Hallaj et al. [8] conducted an extensive investigation into using phase change materials in lithium-ion battery applications. In a research conducted by Ho and Gao [9], nonionic surfactants were used to disperse Al_2O_3 nanoparticle into phase change materials, and thermo-physical properties of the nanoparticle-embedded PCM were experimentally investigated. Dong et al. [10] used electrospinning to synthesize nanofibers of poly (vinyl pyrrolidone) and poly (vinyl alcohol). Teng and Yu [11] combined various nanoparticles with PCMs to synthesize nanocomposite enhanced phase change materials (NEPCMs). Wang et al. [12] studied the effects of addition of titanium dioxide nanoparticles to the paraffin waxes on the thermal properties of PCM. Lee et al. [13] coated a thin layer of aluminum oxide nanoparticles on polyimide electrospun membranes of poly (amic acid) to make nanofibrous separators. These nanofibrous separators showed an enhancement in the capacity and a slight increase in cell impedance. Cai et al. [14] synthesized stable phase change material with nanofiber composites by electrospinning silicon dioxide in capric-lauric-palmitic acid for thermal energy storage applications. Heat transfer properties of expanded perlite composite modified by graphite and capric myristic acid were studied by Karaipekli and Sari [15]. Wu et al. [16] used copper nanoparticles to synthesize nanofluid PCM, and the resulted PCM decreased the solidification and liquefaction times by 28.2% and 30.3%, respectively. Arasu and Mujumdar [17] performed numerical analysis on phase change behavior of paraffin wax modified with aluminum oxide nanoparticles in a rectangular chamber, and their results showed that the variation of temperature on the heating wall was almost negligible (at 330 K), while other walls were isolated. Their results showed that the vertical wall has a higher melting rate that the horizontal fence. Arsu et al. [18] studied the effect of the addition of aluminum oxide nanoparticles to paraffin. Results of their numerical simulation showed that the thermal conductivity of the Al_2O_3-paraffin nanocomposite was dramatically increased compared to the blank paraffin. Xu and Li [19] fabricated stable composites of paraffin modified by diatomite and multi-walled carbon nanotubes (MWCNT) for TES applications. They claimed that in their composite, the thermal conductivity and heat storage rate has greatly enhanced compared to the blank paraffin, and thermal conductivity was enhanced up to 43% after addition of 0.26% of MWCNT. Ramakrishnan et al. [20] developed a novel phase change material composite with the base of hydrophobic expanded perlite, which has applications in construction materials. Babapoor et al. [2] studied the effect of addition of different types of metal oxide nanoparticles to paraffin on the thermal behavior of PCM. They improved the thermal conductivity of paraffin by applying highly thermal conductive nanoparticles in PCM nanocomposites. Despite all improvements in thermal storage systems, to the best of our knowledge, there is no comprehensive study about the performance of composites containing nano-graphene and metal nanoparticles. This study compares the performance of different paraffin-based nanocomposites modified by graphene and metal nanoparticles. Various analysis methods, such as scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FT-IR), and differen-

tial scanning calorimetry (DSC) were performed to study the thermal performance of nanocomposites. Because of the unique characteristics of NEPCM composites, such as high thermal stability, low price, and chemical stability after large numbers of cycles, they have many potential applications in thermal management and storage industries.

2. EXPERIMENTAL

A. Materials
The PCM nanocomposites were synthesized according to the literature [21] with some modifications. Solid and liquid paraffin were mixed to achieve the melting range of 53-57 °C. Sodium dodecyl sulfate (SDS) and nanoparticles of copper (II) oxide, aluminum oxide, titanium (IV) oxide, and graphene were purchased from Merck. Tables 1 and 2 represent the thermophysical characteristics of these materials.

B. Shape-stabilized PCM synthesis
The PCM was synthesized according to the literature [21] with some modifications. Firstly, the base PCM was prepared by mixing solid and liquid paraffin to achieve the desired melting point (53-57 °C) for thermal management purposes. Solid paraffin was heated to a temperature slightly above its melting point and then mixed with the required weight ratio of liquid paraffin to attain the target melting point. Nanoparticles, SDS and paraffin were blended with the required ratio and mixed thoroughly for 120 min (100 rpm). Nanoparticles were dispersed in the composites by sonication at the frequency of 22 kHz at a temperature slightly higher than the melting point to achieve a homogeneous composite. The composite was cooled to room temperature for further use. Table 3 shows the list of synthesized nanocomposites.

C. Characterization of samples
In this study, the dispersion of nanoparticles in the paraffin mixtures and morphological properties of the PCM composites were characterized using SEM (resolution: 5 nm). FTIR analysis (spectra range: 400-4000 cm^{-1}, wavenumber accuracy: 0.1 cm^{-1}) was used to study the chemical structure and verification of nanocomposite formation. Thermal properties of the PCM composites were evaluated by differential scanning calorimetry (DSC). The rate of temperature increase in DSC analysis was 1 °C/min, during both heating and cooling. DSC analysis was initiated at -20 °C, the sample was kept at -20 °C for 5 minutes to stabilize the initial condition, then heated up to 100 C with the rate of 1 °C/min, kept at the constant temperature for 5 minutes, then cooled down with the same rate. The effective thermal conductivity of the NEPCM samples was measured by a digital conductivity meter (manufactured by Sahand Co., temperature accuracy: ± 0.01 °C, absolute thermal conductivity accuracy: ± 3%.) based upon the transient hot-wire method. Thermal conductivity was evaluated at various parts of PCM composites, and the average values and standard deviations are reported.

3. RESULTS AND DISCUSSION

A. Structural morphology of PCM composites
Fig. 1 shows the SEM images of different types of nanoparticles and nanocomposites. Nanocomposites images represent the proper distribution of nanoparticles within paraffin. The heat transfer performance of PCM is expected to be improved by these conductive connecting networks. At higher resolutions,

Table 1. Thermo-physical properties of the organic paraffin

Thermo-physical properties	Units	Magnitude
Melting/ Solidification temperature	K	300.7
Latent heat fusion	kJ/kg	206
Thermal conductivity S-L	W/m K	0.18-0.19
Specific heat S-L	kJ/kg K	1.8-2.4
Density	kg/m^3	789-750

Table 2. Thermo-physical properties of nanoparticles

Nanoparticles	CuO	Graphene oxide nanoparticle	Al$_2$O$_3$	TiO$_2$
Purity	99%	99.50%	99%	99%
Colour	Black	Black	White	White
Particle size	< 50 nm:(APS)	4-20 nm	80 nm: (APS)	20 nm:(APS)
Specific surface area (m^2/g)	> 80 (m^2/g):(SSA)	Diameter: 5-10 μm	< 10 (m^2/g):(SSA)	10-45 (m^2/g):(SSA)
Morphology	Nearly spherical	-	Nearly spherical	Nearly spherical
Bulk density (g/cm^3)	0.79	-	0.79	-
True density (g/cm^3)	6.4	1 (20 ^{0}C)	6.4	-
Resistance	-	4×10^{-4}	-	-
Number of layer	-	< 30	-	-

Table 3. The list of synthesized nanocomposites

Samples	Nanocomposites	Concentration of nanoparticles (wt%)
P	Paraffin (base)	-
Cu-1	Paraffin + CuO + SDS	1%
Cu-2	Paraffin + CuO + SDS	2%
Cu-3	Paraffin + CuO + SDS	3%
Ti-1	Paraffin + TiO$_2$ + SDS	1%
Ti-2	Paraffin + TiO$_2$ + SDS	2%
Ti-3	Paraffin + TiO$_2$ + SDS	3%
Al-1	Paraffin + Al$_2$O$_3$ + SDS	1%
Al-2	Paraffin + Al$_2$O$_3$ + SDS	2%
Al-3	Paraffin + Al$_2$O$_3$ + SDS	3%
G-1	Paraffin + Graphene + SDS	1%
G-2	Paraffin + Graphene + SDS	2%
G-3	Paraffin + Graphene + SDS	3%

single particles are vividly visible in the micrograph and seem to be fully dispersed in the PCM. The high specific surface area of these particles increases their surface activity. Therefore, the increased interfacial adhesion between these nanoparticles and paraffin affects the heat transfer mechanism in the composites.

B. Thermal analysis

Thermal storage is designed to collect energy during melting. Thus, one of the parameters that must be met is the charge time, defined as the amount of time required for energy storage in a TES system. In PCM-based TES systems, the melting process is triggered by the heat flow into the PCM. Since the PCMs state is

mainly solid, the heat transfer is initially observed in the form of conduction. When it begins to melt, in the melted part, heat transfer is mostly due to the natural convection. When charging, the temperature of the PCM increased until it starts to melt. The heat transfer is dominated by conduction from the beginning of the heating process. Subsequently, there is a transition period where conduction and convection occur simultaneously. Then, the melting process continues, and the heat transfer is dominated by convection [22]. DSC results related to pure paraffin (blank sample) in the temperature range of -15 °C to 110 °C are presented in Fig. 2. The lower curve is related to the melting process (forward curve) where the PCM absorbs and stores ther-
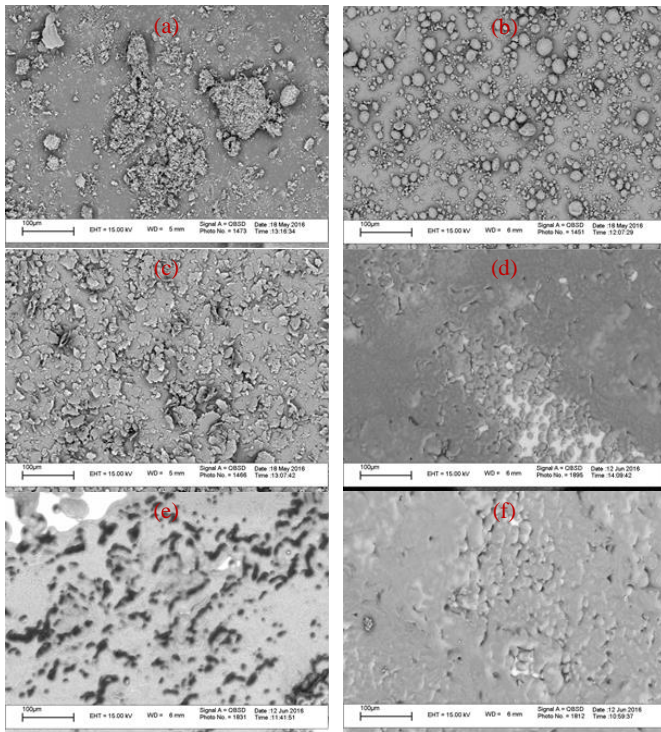
Fig. 1. SEM images of nanoparticles and nanocomposites, (a): pure *CuO* nanoparticle, (b): pure *Al₂O₃* nanoparticle, (c): pure graphene nanoparticle, (d): 3 wt.% *TiO₂* nanocomposite, (e): 1 wt.% *Al₂O₃* nanocomposite, (f): 2 wt.% *CuO* nanocomposite.
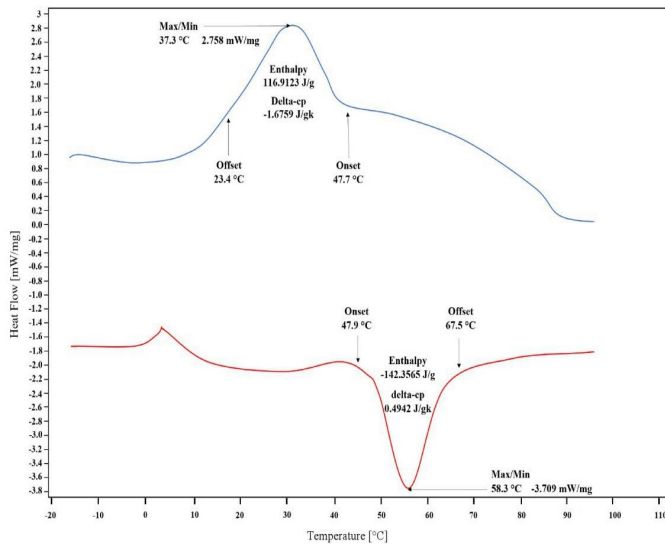

Fig. 2. DSC results related to pure paraffin.

Fig. 3. Paraffin and nanocomposite phase change temperatures in the melting process.

mal energy, and the upper curve is associated with the freezing process (backward) where the PCM emits its stored energy to the ambient. Dominant peaks in the DSC thermograms denote the solid-liquid phase change temperature.

Fig. 3 shows the melting temperatures of NEPCM nanocomposites. The nanocomposites containing 2 wt.% TiO₂ (T_{onset} = 50.2 °C) and 2wt.% Al₂O₃ (T_{onset} = 40.2 °C) had the highest and the lowest onset temperatures, respectively; while the pure paraffin had the onset temperature of T_{onset} = 47.9 °C. However,
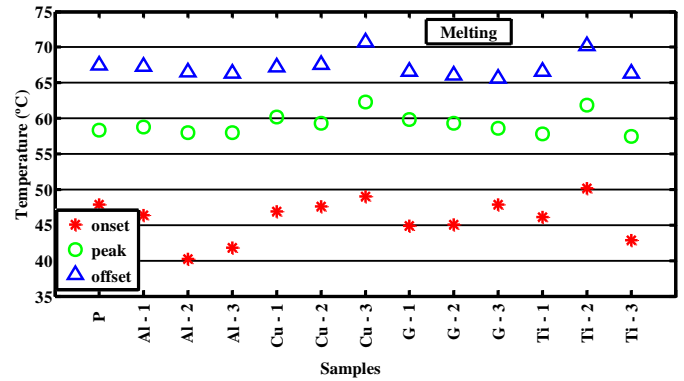
the nanocomposites containing 3 wt.% CuO (T_{peak} = 62.3 °C) and 3 wt.% TiO₂ (T_{peak} = 57.5 °C) had the highest and the lowest peak temperatures, respectively. The reason for the increase in onset temperature is the increase in the percentage of nanoparticles that originates from the proper distribution and appropriate size of nanoparticles. It was observed in the melting process that samples of 3 wt.% CuO nanocomposite (T_{offset} = 70.7 °C) and 2 wt.% graphene nanocomposite (T_{offset} = 66.1 °C) had the highest and the lowest offset temperatures respectively, relative to paraffin (T_{offset} = 47.9 °C). Although paraffin has a relatively high thermal capacity, it suffers from low thermal conductivity [23–26]. However, the addition of nanoparticles to paraffin causes an increase or decrease in the onset and the offset temperatures. Another parameter to take into consideration is the weight percent of nanoparticles [26]. The nanocomposite with 1 wt.% Al₂O₃ had a higher onset temperature (T_{onset} = 46.4 ^0C) compared to those with 2 and 3 wt.% Al₂O₃ which had onset temperatures of 40.2 and 41.8 °C, respectively. Moreover, the composite with 1 wt.% Al₂O₃ had the highest peak temperature at 58.8 °C compared to the composites with 2 wt.% Al₂O₃ (58 °C) and 3 wt.% Al₂O₃ (58 °C), respectively. According to the Fig. 3, it is observed that the nanocomposite with 3 wt.% graphene had the highest onset temperature at 47.9 °C compared to the composites with 2 and 1 wt.% at 45.1 and 44.9 °C, respectively. The nanocomposite with 1 wt.% graphene had the highest offset temperature at 65.6 °C, while the nanocomposites with 2 and 3 wt.% graphene had the offset temperatures of 66.1 °C and 65.6 °C, respectively. Moreover, the nanocomposite with 1 wt.% graphene had the highest peak temperature at 59.8 °C compared to the nanocomposites with 2 and 3 wt.% graphene at 59.3 °C and 58.6 °C, respectively.

Onset and offset temperature difference between nanocomposites and blank paraffin in the melting process is shown in Fig. 4. The highest temperature difference was observed for the nanocomposite with 2 wt.% *Al₂O₃* at 26.3 °C, while the lowest temperature difference was observed for the nanocomposite with 3 wt.% graphene at 17.7 °C in comparison to the blank at 19.6 °C. Obviously, these nanocomposites can improve the performance of thermal management systems. However, in some cases, the low-temperature difference is preferable.

Fig. 5 shows the latent heat (enthalpy) of paraffin and nanocomposites in the melting process. Proper phase change materials possess large latent heat values to make them suitable for thermal storage applications. The addition of thermally conductive fillers to the PCM results in lower values of latent heat
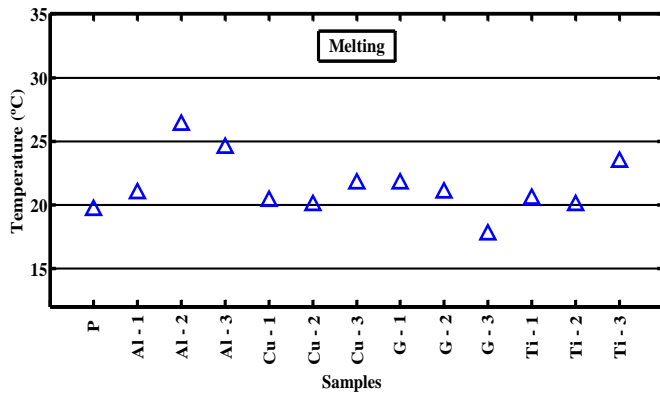
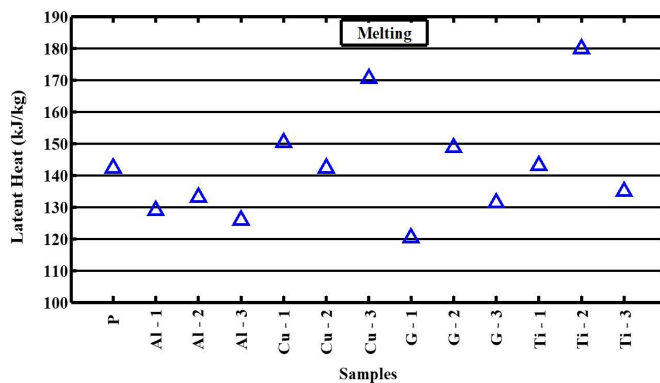**Fig. 4.** Onset and offset temperature difference in the melting process.

Fig. 5. Phase change latent heat of paraffin and nanocomposites in the melting process.

Fig. 6. Thermal conductivity coefficient of paraffin and nanocomposites.

Fig. 7. FTIR spectra of these materials.

compared to the blank PCM. Therefore, it is a must to make the right balance between the increase in the heat transfer coefficient and latent heat reduction. Consequently, it is better to use fillers with high specific surface areas. In the melting process (Fig. 5), comparing the enthalpy of nanocomposites with paraffin indicates that the nanocomposite with 2 wt.% TiO_2 with enthalpy of 179.8846 J/g and the nanocomposite with 1 wt.% graphene with the enthalpy of 120.3800 J/g have the highest and the lowest energy storage in comparison to the blank paraffin with an enthalpy of 142.3565 J/g. Typically, the latent heat of PCM composite decreases by adding nanoparticles due to the molecular interactions between nanoparticles and PCM molecules. Thus, an optimal composite was chosen considering the lowest enthalpy reduction and the highest thermal conductivity enhancement.

C. Thermal conductivity coefficient

The thermal conductivity coefficient of paraffin and nanocomposites is shown in Fig. 6. Results show that 3 wt.% graphene nanocomposite has the highest thermal conductivity coefficient and 1 wt.% TiO_2 nanocomposite has the lowest thermal conductivity coefficient among nanocomposites. It was observed that the increase in the percentage of nanoparticles resulted in the enhancement of the thermal conductivity coefficient. Many parameters, such as distribution and type of nanoparticles, affect the thermal conductivity coefficient. Experimental results show that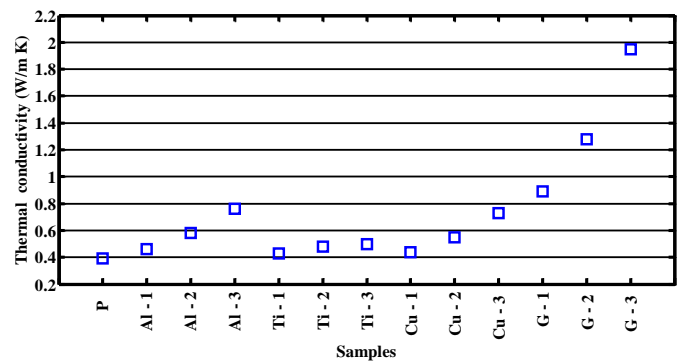 using optimal amounts of nanoparticle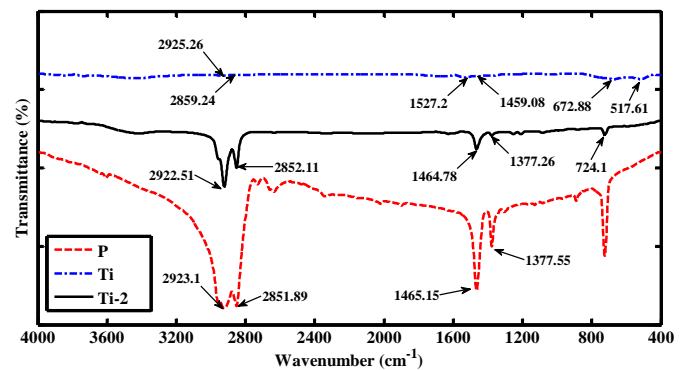s has a crucial role in the enhancement of the conductivity of nanocomposites [2]. Graphene nanoparticle has a high thermal conductivity; therefore, they showed the highest effect on the enhancement of effective thermal conductivity of nanocomposites. Accordingly, graphene nanocomposites are suggested in the cases that the main aim is to have a high thermal conductivity.

D. FTIR analysis

Fig. 7 compares the FTIR spectra of paraffin, TiO_2 nanoparticles and Ti-2-nanocomposite. Spectra peaks of paraffin at 2845 cm^{-1} and 2915 cm^{-1} are due to the stretching vibration of CH bond and the peaks at 1375 cm^{-1}, and 1455 cm^{-1} are due to bending vibration of CH_2 bond [27]. For TiO_2 nanoparticles, the peak at 725 cm^{-1} is relevant to TiO_2 stretching vibration [28]. Peaks at 2852.11 cm^{-1} and 2922.51 cm^{-1} in the nanocomposite 2 wt.% TiO_2 are related to paraffin and peak at 724.10 cm^{-1} are contributed to Ti-O tensile vibration. The spectra obtained from composite are similar to paraffin and TiO_2 nanoparticle, which indicates the formation of the composite. The FTIR results showed that no chemical reaction occurs between the blank paraffin and nanoparticles, and the NEPCM is the result of the dispersion of nanoparticles in paraffin.

4. CONCLUSION

In conclusion, we synthesized stable forms of phase change material nanocomposites by the combination of paraffin as the PCM with either CuO, Al_2O_3, TiO_2 or graphene nanoparticles in the presence of sodium dodecyl sulfate (SDS) as a surfactant. Thermal properties, morphological structure, and forma-

tion of nanocomposites were investigated by DSC, SEM, FT-IR tests. SEM results showed that nanoparticles were uniformly distributed in the PCM. Comparing the enthalpy of nanocomposites during melting process showed that the nanocomposite with 2 wt.% TiO$_2$ with enthalpy of 179.8846 J/g and the nanocomposite with 1 wt.% graphene with an enthalpy of 120.3800 J/g had the highest and the lowest energy storage capacity relative to the blank paraffin with an enthalpy of 142.3565 J/g. Mostly, the onset temperature was decreased in the forward curve due to the high thermal conductivity coefficient of nanoparticles. According to Fourier law ($q = -k\frac{dT}{dx}$), in this case, q is constant, and the temperature changes ($\frac{dT}{dx}$) decreases. To summarize, adding nanoparticles to the paraffin decreases the onset temperature due to the thermal conductivity of nanoparticles; however, it reduces the latent heat of PCM composites at the same time. Therefore, the optimum condition was incorporated to balance out the thermal conductivity improvement and latent heat loss. The experimental results demonstrated the thermal properties such as heat transfer rate and thermal energy storage capacity of NEPCM composites were notably improved compared with the blank PCM with no additives.

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